

EXPLOSIVE RESIDUES
STANDARD OPERATING PROCEDURE
U.S.EPA Region II

Date: Sept., 1994
Revision: 1.3

YES NO N/A

CASE NUMBER: _____

LAB: _____

SITE: _____

1.0 Introduction

- 1.1 The attached Standard Operating Procedure (SOP) is applicable to nitro substituted aromatics and nitro substituted amines by High Performance Liquid Chromatography (HPLC) data. Its scope is not only to facilitate the data validation process of the data reported by the contracting laboratory but also to ensure that the data is being reviewed in a uniform manner.
- 1.2 The SOP is based upon the quality control and quality assurance requirement specified in the analytical **Method 8330, November 1992 (Revision 0).**

2.0 Responsibilities

- 2.1 The reviewer must be knowledgeable of the analytical method and its QC criteria.
- 2.2 The reviewer must complete and/or file the following:

Data Assessment Checklist- The data reviewer evaluates each criterion carefully and checks if data is in compliance, non-compliance or not applicable.

Data Assessment Narrative- The data reviewer must present professional judgement, address areas of concern and comment on the validity of the overall data package. The reviewer must explain the reasons for rejecting and/or qualifying the data.

Rejection Summary Form- The reviewer must submit a completed form using a ratio format.

Organic Regional Data Assessment Summary- A completed form must be submitted.

Telephone Record Log- All phone conversations must be transcribed by the reviewer. Upon completion of

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the data review, the original telephone log
is attached to the data assessment narrative.

3.0 Data Completeness and Deliverables

3.1 Have any missing deliverables been received
and added to the data package? ☐ ☐ ☐

ACTION: Call lab for explanation/resubmittal of
any missing deliverables. If lab cannot
provide them, note the effect on review of
the package under the "Contract Problems/Non-
Compliance" section of reviewer narrative.

3.2 Was SAS-request included with package? ☐ ☐ ☐

If no, a SAS-request can be retrieved from RSCC.

4.0 Cover Letter SDG Narrative

4.1 Is the Narrative or Cover Letter Present? ☐ ☐ ☐

4.2 Are Case Number and/or SAS number contained
in the Narrative or Cover letter? ☐ ☐ ☐

5.0 Traffic Reports and Laboratory Narrative

5.1 Are Traffic Report Forms present for
all samples? ☐ ☐ ☐

ACTION: If no, contact lab for replacement of
missing or illegible copies.

5.2 Do Traffic Reports or Lab Narrative
indicate any problems with sample receipt,
condition of samples, analytical problems
or special circumstances affecting the
quality of the data? ☐ ☐ ☐

NOTE: Samples are not preserved.

ACTION: If samples were not iced upon receipt at
the laboratory, flag all positive results
"J" and all non-detects "UJ".

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NOTE: Samples must be kept below 4°C from the time of collection through analysis. Degradation of some compounds occurs with heat. Samples should not be exposed to sunlight or temperatures above room temperature.

6.0 Special QC

6.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water should be analyzed to determine possible interferences with analyte peaks. A different batch of solvent should be used if contamination is present.

6.2 Chromatograms are to be submitted showing that there are no interferences with analyte peaks.

Are these chromatograms present in package? ☐ ☐ ☐

Are the chromatograms free of interferences? ☐ ☐ ☐

ACTION: Ask lab for resubmittals. If deliverables are unavailable, judge the effect on the validity of the data. If questionable, contact SMO and note in data assessment.

7.0 Holding Times

7.1 Have any technical holding times, determined from date of collection to date of analysis, been exceeded?

A. For aqueous samples 7 days from sample collection to extraction ☐ ☐ ☐

B. For soil/sediment samples 14 days from sample collection to extraction ☐ ☐ ☐

C. For all samples extracts 40 days from time of extraction to time of analysis ☐ ☐ ☐

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Holding Time Violations

Table 1

(See Traffic Report)

Sample ID	Date Sampled	Date Received	Date Lab Extracted	Date Analyzed
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded. The reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J", but the reviewer may determine that non-detect data are unusable (R).

8.0 Surrogate Recovery

8.1 Are the Organic Analysis Data Sheets (Form I) present and complete with surrogate recoveries for each of the following matrices:

- a. Water? [] ____
b. Soil/Solid? [] ____

ACTION: Call lab for explanation/resubmittals.
If missing deliverables or information are unavailable document the effect in the data assessment.

8.2 Was the surrogate % recovery out of specifications for any field sample, method blank or QC sample? ____ [] ____

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YES NO N/A

8.3 Were surrogate retention times (RT) within
± 1.0% of the mean surrogate RT calculated
from the initial calibration?

☐ ☐ ☐

ACTION: Circle outliers in red.

ACTION: If the retention time shift (RTS) or % recovery
of the surrogate in the field or QC samples is out of
specification, then all associated sample data
should be qualified. Use **Table 2** below as a guide.

Surrogate Recovery

TABLE 2

The following table summarizes the surrogate recovery criteria and the data qualification
guidelines for all associated field samples.

SURROGATE	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	50 - 125%	< 50%; > 125%		
Non-detects	10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	50 - 125%	< 50%; > 125%		
Non-detects	20%	10 - 19%	< 10%	
RTS - field samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RTS - QC samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

** If the surrogate recovery in a QC sample or blank was less than 50 percent or
greater than 125 percent, then the field sample data should be qualified if the surrogate in
the field sample is outside 50 - 125 percent recovery.

8.4 Are there any transcription/calculation errors
between raw data and surrogate recoveries on Forms: ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/
resubmittal, make necessary corrections and note
errors in the data assessment.

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9.0 QC Check Reference Sample

9.1 Is the QC Check Reference Sample Recovery Form present? (Created by Lab)

☐ ☐ ☐

9.2 Was the QC Check Reference Sample analyzed at the required frequency (once per SDG or every 20 samples, for each matrix?)

☐ ☐ ☐

NOTE: The QC Check Form has to be created by the Lab. Concentration of the spiking solution is 5-10 times the estimated quantitation limits for all analytes listed in section 12 of the SAS.

NOTE: QC Check Reference Sample information is important, data is used to judge extraction efficiency.

ACTION: If any QC Check data are missing, call the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, document the effect on the validity of the data in the data assessment. Positive hits should be flagged "J" and non-detects should be flagged "R".

9.3 How many QC check recoveries are outside QC limits of 70-125%?

Water

Soil

_____ out of ____

_____ out of ____

ACTION: If any recovery is greater than 125%, positive results should be flagged "J" for the affected compound.

When two or more analytes have recoveries above 10% but below 70% all associated data should be flagged "J" (positive and non-detects).

When one or more analytes have recoveries below 10% qualify all associated data "R".

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Note in data assessment. If further assistance is required, contact SMO for instructions.

It should be noted for TPO action, if a laboratory fails to analyze a QC Check Reference Sample or if a laboratory consistently fails to generate acceptable recoveries.

10.0 Calibration Data

10.1 Initial Calibration

The initial calibration standards must be analyzed daily prior to any sample analysis. The lab may have to create two sets of initial standard solutions if several analytes coelute.

The initial calibration curve must be injected in triplicate for each of the 5 levels.

10.2 Are the chromatograms and data system printouts (Quant reports) present for initial and triplicate calibrations?

☐ ☐ ☐

10.3 Are the modified Initial Calibration Forms (Pest VI-1, VI-2 Forms) present?

☐ ☐ ☐

10.4 Are modified Forms present and completed for each analytical sequence and each column?

☐ ☐ ☐

ACTION: If any Calibration Standard chromatograms and forms are missing, contact the lab for resubmittals.

10.5 Are there any transcription/calculation errors between raw data and Forms?

☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal. Make necessary corrections and document effect in data assessment. Check at least 2-3 values from each calibration standard and more if errors are found.

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YES NO N/A

10.6 Do all standard retention times, including each compound in each level, fall within the windows established during the initial calibration analytical sequence?

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NOTE: All injections are taken into account: A total of 15 injections (three injections for each of the five levels). If the lab had to run two sets of standards there would be a total of 30 injections.

ACTION: The average retention time (RT) for each analyte is determined from the mean of the triplicate injection for each 5 point level of the initial calibration. The RT window for each analyte is equal to the average RT \pm three (3) times the standard deviation. If the standard deviation comes out to equal zero, the window will be ± 0.08 . The (Pest form VI-1) Form should be modified to include the average of the three injections for each of the 5 levels.

10.7 Are the linearity criteria for the initial analyses within limits for both columns? (%RSD must be $\leq 20\%$)

☐ ☒ ☐

ACTION: If no, qualify all associated positive results generated during the sequence "J" and all non-detects "UJ". When %RSD $> 90\%$, flag all non-detects results for that analyte as unusable "R".

10.8 Do the standards show degradation products of teteryl?

☐ ☒ ☐

NOTE: Degradation products of teteryl appear as a shoulder on the TNT peak. Peak heights rather than peak areas should be used when teteryl is present in concentrations that are significant relative to the concentration of TNT.

ACTION: If degradation is detected or suspected, qualify positive hits for teteryl and TNT as "J". If degradation is suspected, and teteryl is reported as non-detected,

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qualify the analyte as "UJ". Note in
data assessment.

11.0 Continuing Calibration

11.1 Are Forms (Modified Pest VII-2) present for
each continuing calibration?

☐ ☐ ☐

11.2 Are there any transcription/calculation
errors between raw data and forms?

☐ ☐ ☐

ACTION: If large errors exist, call lab for
explanation/resubmittals. Make any
necessary corrections, and document
effect in data assessment. Check at
least 2-3 values from each calibration
standard and more if errors are found.

11.3 Were continuing calibrations analyzed
at the required frequency? (standard
should bracket every 10 samples).

☐ ☐ ☐

ACTION: Criteria must be met. Determine
effect on data. At a minimum, all
data should be qualified "J", but the
reviewer may determine that data
are unusable "R". Document the data
qualifications in data assessment.

11.4 Do all standard retention times for
each continuing calibration fall
within the windows established by
the initial calibration?

☐ ☐ ☐

ACTION: If no, beginning with the samples which followed
the last in-control standard, check to see if
the chromatograms contain interfering peaks.
If peaks are present, qualify positive results
and non-detects for analytes outside their respective
windows as unusable "R".

11.5 Are the RPD values for all continuing
calibration standards $\leq 15\%$?

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ACTION: If the %RPD is >15% for the analyte being quantitated, qualify all associated positive results "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard. If the %RPD is >90%, flag all non-detects for that analyte unusable "R".

11.6 Compare the peak heights of continuing calibration checks obtained during the day with peak heights obtained from first calibration check of the sequence.

Is the percent RPD for peak heights less than 20% for each of the analytes of each of the Continuing Calibration Checks? ☐ ☐ ☐

ACTION: Use professional judgement to verify the reliability of the data.

12.0 Analytical Sequence

12.1 Is Form VIII present and completed for each column and each period of analyses? ☐ ☐ ☐

ACTION: If no, contact lab for resubmittals.

12.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? ☐ ☐ ☐

NOTE: Sequence is as follows: 5 point Initial Calibration, Method Blank, Laboratory Control Sample (LCS), Continuing Calibration, 10 sample extracts, Continuing Calibration, 10 sample extracts and so on. The sequence must always end with a Continuing Calibration. All sample extracts, Method Blanks, LCS, and continuing calibrations must be analyzed within 24 hours after the analysis of the Initial Calibration Standards and the calculation of the RT windows.

ACTION: Flag "J" all data generated outside an acceptable twenty-four hour sequence starting after the calculation of the RT windows resulting from the

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initial calibration unless the daily calibration meets the calibration criteria for all target analytes. If the daily calibration standard does not meet the criteria for some analytes the reviewer might reject

those values. In any case, if the 24 hour sequence is exceeded it should be noted in the data assessment under contract non-compliance.

13.0 Method Blanks

13.1 Is the Method Blank Summary (Form IV) present for each of the following matrices?

- a. water
- b. soil

☐ ☐ ☐

13.2 Frequency of Analysis:

Has a Method/Prep blank been analyzed at the same time for each batch of samples extracted.

☐ ☐ ☐

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If method blank data are not available, reject "R" all associated positive data.

13.3 Are the positive hits present in the blank less than detection limit?

☐ ☐ ☐

ACTION: If hits are \geq detection limits, analysis should be discontinued. All associated samples must be re-prepared and re-analyzed with a new blank. If lab did not follow this action, note in data assessment. Follow guidelines under contamination section.

13.4 Chromatography: review the method blank raw data chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable?

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YES NO N/A

ACTION: Use professional judgement to determine the effect on the data.

14.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

14.1 Did the lab run an instrument blank following a sample analysis which contained an analyte(s) at high concentration(s) to evaluate possible sample cross-contamination?

☐ ☐ ☐

ACTION: Sample analysis results after the high concentration sample must be evaluated for carryover. Sample cross-contamination should be noted for TPO action if an effect on the data is suspected. An Instrument Blank is not required in the methodology.

14.2 Do any Method Blanks have positive results (TCL)? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor.

☐ ☐ ☐

14.3 Are there field/rinse/equipment blanks associated with every sample?

☐ ☐ ☐

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

14.4 Do any field/rinse blanks have positive results (TCL)?

☐ ☐ ☐

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data.

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Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate recovery, instrument performance criteria, or calibration QC problems.

ACTION: Follow the directions in **Table 3** below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated data should be qualified as unusable (R) (see item # 12.3).

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration or other QC criteria.

15.0 Target Compound List (TCL) Analytes (Form I)

15.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

- | | | | |
|---|--------------------------|-----|-----|
| a. Samples ? | <input type="checkbox"/> | ___ | ___ |
| b. Laboratory Control Samples? | <input type="checkbox"/> | ___ | ___ |
| c. Blanks? | <input type="checkbox"/> | ___ | ___ |
| d. Matrix spikes and matrix spike duplicates? | <input type="checkbox"/> | ___ | ___ |
| e. Lab duplicate? | <input type="checkbox"/> | ___ | ___ |

15.2 Are the Chromatograms, and the data system printouts (Quant Reports) included in the sample package for each of the following:

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- | | | | |
|--|--------------------------|-----|-----|
| a. Samples? | <input type="checkbox"/> | ___ | ___ |
| b. Laboratory Control Samples? | <input type="checkbox"/> | ___ | ___ |
| c. Blanks? | <input type="checkbox"/> | ___ | ___ |
| d. Matrix spikes and Matrix spike
duplicates? | <input type="checkbox"/> | ___ | ___ |
| e. Lab duplicate? | <input type="checkbox"/> | ___ | ___ |

ACTION: If any data are missing, Contact
Lab for resubmittals.

15.3 Are the response factors shown in the Quant
Report? ☐ ___ ___

15.4 Is chromatographic performance acceptable
with respect to:

Baseline stability?	<input type="checkbox"/>	___	___
Resolution?	<input type="checkbox"/>	___	___
Peak shape?	<input type="checkbox"/>	___	___
Full-scale graph (attenuation)?	<input type="checkbox"/>	___	___
Other: _____	<input type="checkbox"/>	___	___

ACTION: Use professional judgement to determine
the acceptability of the data.

16.0 Compound Identification

16.1 Is Form X completed for every sample in which
an analyte was detected? ☐ ___ ___

ACTION: If no, contact lab for resubmittals.

16.2 Are there any transcription/calculation errors
between raw data and Forms? ___ ☐ ___

ACTION: If large errors exist, call lab for
explanation/resubmittals. Make any
necessary corrections and note effect
in data assessment. Check at least 2-3
values from each calibration standard
and more if errors are found.

16.3 Are retention times (RT) of sample analytes
within the established RT windows for both

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analyses?

☐ ☐ ☐

ACTION: Qualify as unusable "R" all positive results which were not confirmed by second HPLC column analysis. Also qualify as unusable "R" all positive results not meeting RT window unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

16.4 Is the percent difference (%D) calculated for the positive sample results on the two HPLC columns <25%?

☐ ☐ ☐

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

<u>% Difference</u>	<u>Qualifier</u>
25-90%	J
>90%	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

16.5 Check chromatograms for false negatives, interferences and degradation. Tetryl decomposes rapidly in methanol/water solutions as well as with heat. All samples expected to contain tetryl should not be exposed to temperatures above room temperature. Degradation products of tetryl appear as a shoulder on the TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration

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YES NO N/A

of TNT.

ACTION: Use professional judgement to determine qualification of analytes.

17.0 Compound Quantitation and Reported Detection Limits

17.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the calculations were adjusted for percent moisture. Were any errors found?

___ ☐ ___

NOTE: Single peak analyte results can be checked for rough agreement between quantitative results obtained on the two HPLC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity "JN". This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has interfered with the evaluation of the second column confirmation.

17.2 Are the CRQLs adjusted to reflect sample dilutions for each Form I?

☐ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note in data assessment.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original

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analysis by crossing out the "E" and its associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" with a red pencil across the entire page of all Form I's that should not be used, including any in the summary package.

18.0 Lab Duplicates

18.1 Were any lab duplicates submitted for
for each extraction batch?

- a. waters
- b. soils

☐ ☐ ☐
☐ ☐ ☐

18.2 Are the Relative Percent Difference (RPD)
values for the analytes reported in the
duplicate samples <20%? (depending on the
SAS-request; the criteria might be <40%).

☐ ☐ ☐

ACTION: Compare the reported results for lab
duplicates and calculate the relative
percent difference.

ACTION: Any gross variation between duplicate
results must be addressed in the reviewer
narrative. However, if large differences
exist, identification of lab duplicates
should be confirmed by contacting the lab.

ACTION: Flag all associated data with an * for
"out of control" duplicate.

19.0 Matrix Spikes

19.1 Is the Matrix Spike/Matrix Spike Duplicate
recovery Form present?

☐ ☐ ☐

19.2 Were the matrix spikes analyzed at the required
frequency? (one per matrix and per SDG).

☐ ☐ ☐

ACTION: If any matrix spike data are missing,
contact the lab for resubmittals.

19.3 The matrix spike consists of all target

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analytes at concentrations 5-10 times the
estimated quantitation limits listed in
Table 1/Method 8330.

Were the correct analytes and were the
concentrations within range?

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NOTE: Field blanks cannot be used for Matrix
spike analysis. If field/rinse blanks were
MS/MSD, then make note under Contract non-compliance.

19.4 How many spike recoveries are outside the
QC limits of 50-140%?

Water

Soil

_____ out of _____

_____ out of _____

19.5 How many RPD's for Matrix Spike and Matrix
Spike duplicate recoveries are outside QC
limits of 20% (depending what is stated in
the SAS-request)?

Water

Soil

_____ out of _____

_____ out of _____

ACTION: Flag all associated data "N" for "out of
control" matrix spike for the field sample
used for the MS/MSD.

ACTION: No additional action is taken on MS/MSD
data alone. However using informed
professional judgement, the data reviewer
may use the matrix spike data results in
conjunction with other QC criteria and
determine the need for some qualification of the data.